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environmental restoration project

A Department of Energy
Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Operation of the Spectrace 9000 Field-Portable X-Ray Fluorescence Instrument

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Revision Log

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Operation of the Spectrace 9000
Field-Portable X-Ray Fluorescence Instrument

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Operation of the Spectrace 9000 Field-Portable X-Ray Fluorescence Instrument

1.0 PURPOSE

This standard operating procedure (SOP) describes the process for operating and using the Spectrace 9000 field-portable x-ray fluorescence (XRF) analyzer to screen for hazardous, or potentially hazardous, inorganic materials at the Los Alamos National Laboratory (Laboratory) ER Project. The data that are generated allow for rapid evaluation of the extent of contamination. Samples can be analyzed for elements of atomic number 13 (aluminum) through 92 (uranium), with proper x-ray source selection and instrument calibration. Environmental applications include measuring elemental metals in soils and on filters and measuring lead in paint.

2.0 SCOPE

This SOP is a mandatory document and shall be implemented by all ER Project participants when conducting XRF field screening on soil and other solid environmental media for the ER Project

3.0 TRAINING

3.1 All users of this QP are trained by reading the procedure. The **user** shall ensure the training is documented in accordance with QP-2.2, Personnel Orientation and Training, and is entered in the ER Project Training Database located at http://erinternal.lanl.gov/Training/Training.asp.

3.2 The **Field Team Leader** (FTL) shall monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

3.3 Personnel using the field-portable XRF instrument must read and follow the instructions found in the manufacturer's operating manual. Personnel may also refer to the Spectrace 9000 Field-Portable/Benchtop XRF training and applications manual and the US Environmental Protection Agency's SOP for the Spectrace 9000 (SOP 1713).

4.0 DEFINITIONS

4.1 *Site-Specific Health and Safety Plan (SSHASP)*—A health and safety plan that is specific to a site or ER-related field activity that has been approved by an ER health and safety representative. This document contains information specific to the project including scope of work, relevant history, descriptions

of hazards by activity associated with the project site(s), and techniques for exposure mitigation (e.g., personal protective equipment [PPE]) and hazard mitigation.

4.2 *X-ray fluorescence spectroscopy*—A nondestructive qualitative and quantitative analytical technique used to determine the elemental composition of solid, liquid, thin film, and powder samples.

5.0 BACKGROUND AND PRECAUTIONS

Note: This SOP is to be used in conjunction with an approved SSHASP. Also, consult the SSHASP for information on and use of all PPE.

5.1 Background

5.1.1 The Spectrace 9000 energy-dispersive field-portable XRF instrument includes three compact sealed radioactive sources: iron (^{55}Fe), cadmium (^{109}Cd), and americium (^{241}Am), which are used for identification and quantitation of 26 elements. The instrument is equipped with a high-resolution HgI_2 detector, which is connected to a sealed electronic module.

5.1.2 The electronic unit contains a 2048 multi-channel analyzer for spectral analysis. It provides internal nonvolatile memory for storage of 120 spectra and results for 300 multi-element analyses, which can be shown on the instrument's display panel. An RS-232C serial port allows users to download data and spectra to a peripheral device such as a printer or IBM-compatible personal computer.

5.1.3 The Spectrace 9000 can be powered by a 115-/220-V wall outlet or by its 4-hr rechargeable battery. The device can be operated in temperatures ranging from 32°F to 120°F. Furthermore, the probe and electronic unit may be exposed to a light rain. Additional protection is provided when the system (electronic unit and probe) is enclosed in the optional water-repellent carrying case. The probe and electronic unit are completely sealed with rubber gaskets and can be decontaminated with soap and water.

5.1.4 The probe battery should be replaced once every 6 months. This requires the top of the probe to be opened. Otherwise, the probe should remain closed and covered with the safety cover, laboratory safety cover, or laboratory safety shield when not in use.

5.2 Precautions

5.2.1 *Instrument integrity*—The Spectrace instrument manufacturer must be notified immediately of any condition or concern relative to the probe's structural integrity, source shielding, source switching

condition, or operability. The appropriate state agency or the US Nuclear Regulatory Commission office must be notified immediately of any damage to the radioactive source or any loss or theft of the device.

5.2.2 *Labels and certifications*—The labels or instructions on the probe(s) must not be altered or removed. The source(s) in the probe must be leak-tested every 6 months, and the leak-test certificates must accompany the instrument at all times.

5.2.3 *Handling*—The Spectrace 9000 should not be dropped or exposed to conditions of excessive shock or vibration. The probe cable must never be pulled while unplugging the probe. The probe plug should be grasped at the ribbed metal connector and squeezed and pulled gently while the connector is unplugged. The connector must never be forcibly plugged in. The handle of the electronic unit must not be rotated unless the release buttons on the side of the handle have been depressed. The Spectrace 9000 should not be stored at an ambient temperature below –4°F or above 110°F.

5.2.4 *Transport*—The Spectrace 9000 must be properly packaged during transport. The suitcase that accompanies the unit is acceptable. DOT requires that the following statement accompany the unit during transportation: “This package conforms to the conditions and limitations specified in 49 CFR 173.422 for acceptable radioactive material, instruments, and articles, UN 2910.”

6.0 RESPONSIBLE PERSONNEL

6.1 Field Team Leader

6.2 Users

6.3 ER Project Participant

6.4 Quality Program Project Leader

6.5 Operator

7.0 EQUIPMENT

The equipment listed below is included with the Spectrace 9000 XRF instrument.

7.1 Analyzer unit for data acquisition, processing, and display

7.2 Hand-held probe

7.3 Three pure element sources (^{55}Fe, ^{109}Cd, ^{241}Am) and a safety cover

7.4 Probe laboratory base stand with safety shield

7.5 Interconnecting cable

7.6 RS-232C serial I/O interface cable and connector

7.7 Blank checks and pure-element check samples

7.8 Spectrace 9000 application software (this software, which is device-specific and not interchangeable, is identified by the serial number of the unit)

7.9 XRF sample cups

7.10 Battery charger

7.11 Instruction manual

7.12 Shipping/carrying case

8.0 PROCEDURE

Note: Subcontractors may follow this standard operating procedure (SOP) for operating the Spectrace 9000 field-portable x-ray fluorescence instrument or may use their own procedure(s) as long as the substitute meets the requirements prescribed by the ER Project Quality Management Plan, and is approved by the Environmental Restoration (ER) Project's Quality Program Project Leader (QPPL) before the commencement of the designated activities.

Note: ER Project participant may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

Note: Personnel must follow the procedures discussed in the manufacturer's operating manual, training manuals, and updates (including Spectrace's "Standard Operating Check" from August 1994). All data must be recorded either in an instrument log book or daily activity log according to instructions in LANL-ER-SOP-01.04, Sample Control and Field Documentation.

8.1 Initial Operation

8.1.1 Connect the battery.

8.1.2 Turn the unit on.

8.1.3 Set the time and date. (It is critical that the date be accurate because the source decay compensation is based on it.)

8.1.4 Allow the Spectrace 9000 to warm up for approximately 30 minutes.

8.1.5 Set desired application (soil, thin filter, lead in paint).

8.1.6 Set measuring times for radioactive sources.

Note: Measuring times depend upon the elements of interest as well as the desired detection limits. Minimum measuring time for each source should be 20 seconds. Although counting statistics improve as measurement time increases, the practical limit for typical applications is 600 to 800 seconds. Refer to Attachment A for the manufacturer's guidance regarding elements excited by radioactive sources and detection limits.

8.1.7 Set time as real or live.

8.1.8 Disable the display threshold.

8.1.9 Set instrument to automatically store results and spectra (optional).

8.2 Probe Use

8.2.1 Make sure the probe is always in contact with the surface of the material being analyzed, and that the material completely covers the probe opening (aperture) when the sources are exposed.

8.2.2 The probe's laboratory safety shield assembly must be used when the probe is inverted for measuring samples contained in cups. Do not remove a sample or move the probe while the message "Source On" is displayed on the screen or while the base of the probe is flashing.

8.2.3 During operation, the probe must be kept at least 3 ft from computer monitors or any other source of radio frequency which could affect measurement results.

8.3 Preoperational Checks

There are several preoperational check samples that should be analyzed prior to sample analysis. The samples include an energy calibration check, a resolution check, a blank sample check, and a target metals response check. (Refer to the Spectrace 9000 training manual and to Spectrace's "Standard Operating Check," August 15, 1994, for detailed procedures on preoperational checks.)

8.3.1 Perform element x-ray response and spectrum resolution check using the Fe blank. This should be performed once at the beginning of each day. Record results in the field or instrument log book.

- If intensities are outside the specified range, refer to Spectrace's "Standard Operating Check." Perform an energy calibration.

- If the Kev value noted in the spectra of the energy calibration check is out of range, refer to the “Standard Operating Check.” An energy calibration may be performed.

8.3.2 Perform analytical background check with the teflon blank at least once a day. Record results in the field or instrument log book.

- If the element concentrations of the elements of concern are greater than five times the standard deviation, refer to the “Standard Operating Check.” The Acquire Background procedure may be performed.

8.3.3 The operator should analyze two standard reference materials (SRMs) [e.g., National Institute of Standards and Technology (NIST) SRM 2710 or 2711] or performance evaluation samples whose metal concentrations bracket the known or suspected metal concentrations of the samples to be measured. If using the instrument for quantitative work, this check must be done at least once per day. Record results in the field or instrument log book to note significant trends in the analytical measurement.

8.3.4 Determine the precision by making multiple analyses of either an SRM (NIST SRM 2710 or 2711) or a thoroughly characterized sample for an evaluation of the precision of the elements of interest. If using the instrument for quantitative work, this must be done at least once per day or once every 20 samples. Conduct this measurement under the same conditions as those used for sample measurements. Record results in the field or instrument log book. The precision objective should be a 20% relative standard deviation.

8.4 Sample Handling and Presentation

The following procedures are recommended for handling and preparing the samples.

8.4.1 Soil Samples Application

Soil samples may be analyzed in either in-situ or in bench-top mode.

8.4.1.1 In-Situ Measurement

- Prepare soil by stirring it with a trowel (or similar utensil).
- Remove rocks, vegetation, or large objects; soils may be sieved.
- Flatten soil with a trowel (or similar utensil).
- Place the probe securely against the flattened soil surface.

- Secure the probe to ensure that it will not be moved or topple over.
- Push either the trigger button on the probe or the CONT button on the instrument to initiate the measurement.

8.4.1.2 Bench-Top Measurement

- Ensure that the sample moisture content is less than that at saturation.
- Prepare the soil until the desired homogeneity is obtained.
- Put the soil into a sample cup and seal it with x-ray film.
- Push the CONT button on the instrument to initiate the measurement.

8.4.2 Thin (Filter) Samples Application

8.4.2.1 Mount the filter and place on the probe. (Filters should be presented loaded-side down and wrinkle-free.)

8.4.2.2 Use the probe safety cover when measuring thin samples.

8.4.2.3 Visually prescreen filters and wipes for defects before using them to establish background and contamination levels.

8.4.3 Lead-in-Paint Application

8.4.3.1 Check that the area selected for analysis is smooth, representative, and free of surface dirt.

8.4.3.2 Hold the Spectrace 9000 probe firmly on the surface.

8.4.3.3 When using the instrument for specimen applications (e.g., on paint chips or nonbacked films), use the probe safety cover.

8.5 Data Quality Assurance (QA)/Quality Control (QC)

A certified reference sample should be run periodically. This will provide valuable accuracy and precision data. Running a certified reference sample can also alert the operator that either the probe window is contaminated or the instrument is not operating properly.

8.6 Recording and Documenting Results

8.6.1 Results (and spectra) may be automatically stored. Refer to the manufacturer's operating manual.

8.6.2 It is recommended that results be downloaded to a personal computer at the end of each day. Refer to the Spectrace 9000 training and applications manual.

8.6.3 Results may be transferred to a spreadsheet.

8.6.4 If data are qualitative (i.e., used only for general surveys or to bias sampling), data may be handled informally and recorded in the field or instrument log book.

8.6.5 ER Project decision data must be transferred to the Facility for Information Management, Analysis, and Display (FIMAD) following peer review. The individual Focus Area's data technician will perform this function. Results for a blank and a check standard should also be reported.

8.7 Perform Lessons Learned

During the performance of work, **ER Project participants** shall identify, document, and submit lessons learned in accordance with QP-3.2, Lessons Learned, located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

9.0 REFERENCES

The following documents have been cited within this procedure.

QP-2.2, Personnel Orientation and Training

QP-4.2, Standard Operating Procedure Development

QP-4.4, Record Transmittal to the Records Processing Facility

QP-5.7, Notebook Documentation for Environmental Restoration Technical Activities

ER-SOP-1.04, Sample Control and Field Documentation

EPA (U.S. Environmental Protection Agency), "Representative Sampling Guidance, Volume 1—Soil," (OSWER Directive 9360.4-10) (EPA Environmental Response Team, Washington, DC, November 1991).

EPA (U.S. Environmental Protection Agency), "Spectrace 9000 Field Portable X-Ray Fluorescence Operating Procedure" (SOP 1713) (EPA Environmental Response Team, Response Engineering and Analytical Contract Standard Operating Procedures, Washington, DC, December 21, 1992).

NIST (National Institute of Standards and Technology), "Standard Reference Material (SRM) 2710, Montana I Soil, Highly Elevated Trace Elements Concentrations, Certification of Analysis," (Standard Reference Materials Program, NIST, Gaithersburg, MD, October 30, 1992).

NIST (National Institute of Standards and Technology), “Standard Reference Material (SRM) 2711, Montana II Soil, Moderately Elevated Trace Elements Concentrations, Certification of Analysis,” (Standard Reference Materials Program, NIST, Gaithersburg, MD, October 30, 1992).

TN Technologies, Inc. (now TN Spectrace), “Spectrace 9000 Portable XRF Analyzer Manufacturers Operator Manual, Part No. 717715, Rev. II,” (TN Technologies, Inc., August 1994).

TN Technologies, Inc. (now TN Spectrace), “Spectrace 9000 Standard Operating Check,” (TN Technologies, Inc., August 15, 1994).

TN Technologies, Inc. (now TN Spectrace), “Spectrace 9000 Field-Portable/Benchtop XRF Training and Applications Manual,” (TN Technologies, Inc., 1994).

Note: TN Spectrace is located at 2555 North Interstate Highway 35, P.O. Box 800, Round Rock, Texas 78680-0800; (800) 736-0801.

10.0 RECORDS

The **Field Team Leader** is responsible for submitting the following records (processed in accordance with QP-4.4, Record Transmittal to the Records Processing Facility) to the Records Processing Facility:

10.1 Completed daily activity log or instrument log book, which should include the following pieces of information:

- Record that the leak test has been performed
- Element X-ray response check
- Analytical background check
- Standards check
- Energy calibration
- Performance evaluation
- Sample preparation
- Measurement time for each radioactive source
- Sample results
- Precision results

11.0 ATTACHMENTS

Attachment A: Elements Excited by Radioactive Sources and Lower Limits of Detection for These Elements (2 pages)

Elements Excited by Radioactive Sources and Lower Limits of Detection for These Elements

For soil samples application using source measuring times of 60 s and 200 s, the following are typical element minimum detection levels (MDLs):

		60 s	200 s
Source	**Element**	**MDL (mg/kg)**	**MDL (mg/kg)**
^{55}Fe	Potassium (K)	325	140
	Calcium (Ca)	150	75
	Titanium (Ti)	110	65
	Chromium (CrLo)	180	117
^{109}Cd	Chromium (CrHi)	525	345
	Manganese (Mn)	410	320
	Iron (Fe)	225	155
	Cobalt (Co)	205	138
	Nickel (Ni)	125	95
	Copper (Cu)	90	55
	Zinc (Zn)	70	50
	Mercury (Hg)	60	50
	Arsenic (As)	50	35
	Selenium (Se)	35	25
	Lead (Pb)	30	20
	Rubidium (Rb)	10	10
	Strontium (Sr)	10	5
	Zirconium (Zr)	10	3
	Molybdenum (Mo)	10	4
^{241}Am	Cadmium (Cd)	180	90
	Tin (Sn)	100	50
	Antimony (Sb)	65	35
	Barium (Ba)	20	10
	Uranium (U)	100	60
	Thorium (Th)	100	60
	Silver (Ag)	100	60

Elements Excited by Radioactive Sources and Lower Limits of Detection for These Elements (continued)

For thin samples application using a source measuring time of 200 s for the ^{55}Fe and ^{109}Cd sources, and 800 s for the ^{241}Am source, the following are typical element MDLs:

Source	Element	MDL (g/cm^2)
^{55}Fe	Potassium (K)	0.40
	Calcium (Ca)	0.20
	Titanium (Ti)	0.15
	Chromium (CrLo)	0.40
^{109}Cd	Chromium (CrHi)	0.90
	Manganese (Mn)	0.65
	Iron (Fe)	0.65
	Cobalt (Co)	0.50
	Nickel (Ni)	0.30
	Copper (Cu)	0.65
	Zinc (Zn)	0.40
	Mercury (Hg)	0.45
	Arsenic (As)	0.40
	Selenium (Se)	0.15
	Lead (Pb)	0.50
	Rubidium (Rb)	0.10
	Strontium (Sr)	0.10
	Zirconium (Zr)	0.15
	Molybdenum (Mo)	0.10
^{241}Am	Cadmium (Cd)	2.5
	Tin (Sn)	2.5
	Antimony (Sb)	1.5
	Barium (Ba)	0.70